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DISSOLVED GASES IN GLASS

BY

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BULLETIN No. 118

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UNIVERSITY OF ILLINOIS
ENGINEERING EXPERIMENT STATION

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DISSOLVED GASES IN GLASS

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DISSOLVED GASES IN GLASS

I. Introduction

1. *Foreword.*—The work described in the following pages was begun in June, 1917, as part of a program of research on some of the problems connected with the manufacture of optical glass. The first experiments were carried out by Mr. Frank F. Footitt, at that time Research Assistant in the Engineering Experiment Station. Mr. Footitt later joined the Signal Corps of the United States Army and was detailed at the University to assist in the continuation of the research. His part in the work continued until he was honorably discharged from the service in February, 1919. The results given in the first three chapters of the present paper are based upon Sgt. Footitt's experiments, an account of which was given before the Pittsburgh meeting of the American Ceramic Society in February, 1919. After Sgt. Footitt's discharge the investigation was dropped until January, 1920, when it was again taken up with the assistance of Dr. Elmer N. Bunting, who is continuing it at the present time.

2. *Purpose of the Investigation.*—All varieties of glass, even at ordinary temperatures, are in the liquid state of aggregation. They are liquids which have been cooled through their normal crystallization interval so rapidly that there has not been time for crystallization ("devitrification") to occur. Instead, the viscosity of the liquid has been increased to such a large value that the molecules do not have sufficient freedom of motion to permit the rearrangements necessary for the formation and growth of crystals. The liquid has thus been supercooled until it has become a solid. In principle any liquid can by supercooling be brought into the condition of a glass, but since it still remains a liquid, it should possess the characteristic properties of liquids, including the power to hold gases in a state of solution.

During the process of manufacturing glass, large quantities of gas, mainly carbon dioxide, oxygen, and nitrogen, are evolved from

the batch owing to the occurrence of chemical reactions such as the following:

$$Na_2CO_3 + SiO_2 = Na_2SiO_3 + CO_2$$
$$4\,KNO_3 + 2\,SiO_2 = 2\,K_2SiO_3 + 2\,N_2 + 5O_2$$
$$2\,PbO_2 + 2\,SiO_2 = 2\,PbSiO_3 + O_2$$

If ammonium nitrate, NH_4NO_3, is employed in "blocking"* the glass, water vapor will also be evolved during the fining. The glass will thus be saturated with these gases at the partial pressures which prevail at the end of the "fining" operation.

On cooling the glass, these gases should remain in solution, and glass in the finished state may therefore be expected to contain appreciable quantities of these dissolved gases. Since no actual data concerning the nature or amounts of such dissolved gases were available, the experiments described below were undertaken for the purpose of throwing some light on this question. These experiments are to be regarded as preliminary to a more extended investigation of these dissolved gases, and of their influence upon the properties of the finished glass, and its behavior during use.

In addition to the account of the experiments conducted to date, and their results, there will be found in the following pages some discussion of the relation between adsorbed and dissolved gas, the influence of dissolved gases upon the properties and behavior of glass, and the use of the vacuum furnace in the manufacture of glass.

3. *Acknowledgments.*—For samples of glass used in the present investigation we are indebted to the United States Bureau of Standards, and to the Pittsburgh Plate Glass Company. The Signal Corps, and later the Aircraft Production Board, made possible the prosecution of the work during the war by the detail of Sgt. Footitt as Research Assistant.

* The term "fining" or "plaining" is applied to the operation of eliminating bubbles from the molten glass. This may be accomplished by heating the glass to a sufficiently high temperature to cause the bubbles to expand and rise to the top of the melt. If this method is not effective the operation of "blocking" is employed. This consists in inserting into the melt, with the aid of an iron rod, a potato, a piece of green wood, a pellet of ammonium nitrate, or in general any material which will give a copious evolution of gas in the form of large bubbles which will rise through the melt and gather up the small bubbles in their path.

Fig. 1. Melting Pot, with Block of Glass before Melting

II. Demonstration of the Existence of Dissolved Gases in Finished Glass

4. *The Method Employed.*—In order to demonstrate the existence of dissolved gases in considerable quantity in a piece of perfectly clear homogeneous glass, the method of "sudden evacuation" may be employed. In this method the piece of glass to be investigated is melted under atmospheric pressure in a vacuum furnace which can be connected through a valve to a large evacuated tank. When the temperature of the glass has reached about 1200 deg. C. the valve is opened quickly, thus causing a sudden drop of pressure within the furnace.

This experiment is similar to the opening of a siphon of soda water, and if the glass contains dissolved gases a similar result would be expected, that is, there should be a sudden evolution of gas from the glass, causing it to expand in volume and to effervesce vigorously.

5. *The Glass.*—The glass employed in the first experiment was a piece of barium flint optical (1.6053-43.6) having the following composition, as determined by the Bureau of Standards:

Oxide . . .	SiO_2	As_2O_5	PbO	ZnO	BaO	K_2O
Mole (per cent)	64.5	0.15	9.73	9.22	10.2	6.24

A piece free from bubbles was selected, placed on a table beside an inverted melting pot, and photographed. (See Fig. 1.)

6. *The Furnace.*—The vacuum furnace and the details of the heating element and thermocouple installation are shown in Figs. 2 and 3, which are self explanatory. The outlet tube was connected to a Nelson rotary vacuum pump and also, through a valve, to a large vacuum tank (A, in Fig. 4) having a capacity about 100 times that of the furnace chamber.

7. *Experimental Procedure.*—The melting pot containing the piece of glass was placed inside the heating chamber (Fig. 2) and this in turn placed within an insulating cylinder supported on the

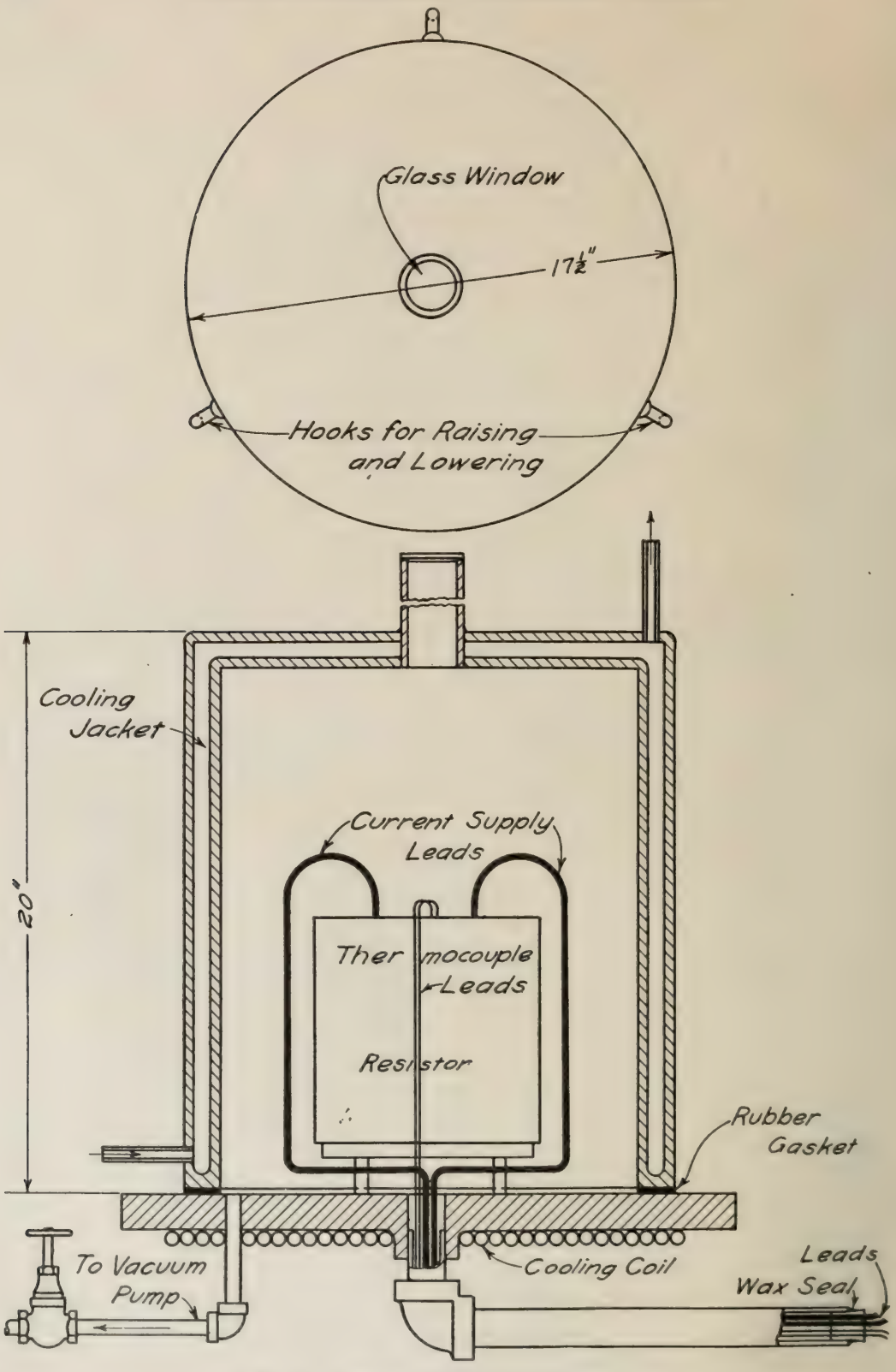

FIG. 2. DETAIL OF VACUUM FURNACE

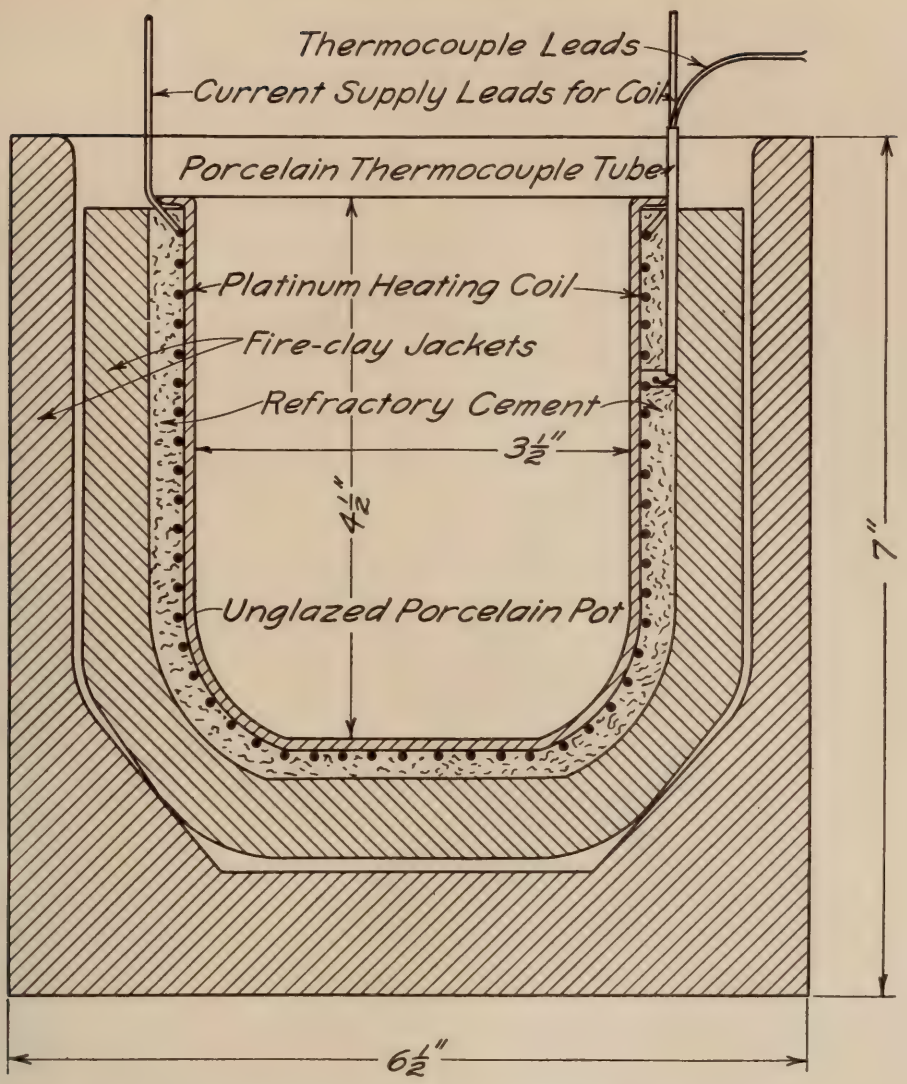

FIG. 3. DETAIL OF POT, RESISTOR, AND INSULATION

furnace base as shown (Fig. 3). The water cooled iron dome (see Fig. 4) was then lowered into place on its rubber gasket and the current started in the heating coil. When the glass had attained a temperature of about 1200 deg. C. the valve connecting the outlet with the large vacuum tank was quickly opened.

This tank had been previously evacuated to a pressure of 1 inch of mercury, and as soon as pressure equalization had taken place, as indicated by the manometer, the valve was quickly closed, the Nelson pump started, and the pressure in the furnace chamber brought down rapidly to less than 1 cm. of mercury. The heating current was then cut off and the furnace allowed to cool with the vacuum on.

8. *The Result.*—On opening the furnace most of the glass was found outside of the pot, standing above it in the form of a large white mass of foam. This was broken away from the pot and photographed as before, beside the inverted pot. The result is shown in Fig. 5. By comparing Figs. 1 and 5 an idea of the increase in volume associated with the evolution of the dissolved gases may be obtained. This amounted to about six times the volume of the original piece. The existence of considerable quantities of gas in a state of solution in the glass was thus demonstrated.

FIG. 4. VACUUM FURNACE AND LARGE VACUUM TANK

FIG. 5. MELTING POT, WITH BLOCK OF GLASS AFTER MELTING AND EVACUATING

III. Partial Analysis of the Gases Evolved from the Glass

9. *The Apparatus and Method.*—The furnace employed was that used in the preceding experiment. The large vacuum tank was disconnected, however, and the outlet tube V was connected to a Gaede high vacuum pump, through an analysing train. The method consisted briefly in evacuating the furnace until all adsorbed gases were removed, melting the glass, drawing the evolved gases out through the analysing train, and finally washing out the furnace with pure nitrogen. All connections throughout the system were sealed glass joints, or glass-to-glass joints covered with heavy rubber tubing and coated with a beeswax-rosin mixture.

10. *The Analysing Train.*—The analysing train consisted of the following elements in the order named, starting from the furnace end:

(1) a series of six gas wash bottles containing standard barium hydroxide solution, and having their delivery tubes drawn down to capillary openings so as to produce a stream of small bubbles through the solution when in operation:

(2) a drying tower containing pumice and sulphuric acid:

(3) a glazed porcelain combustion tube containing copper gauze and provided with a heating coil. Two pieces of copper gauze previously reduced in hydrogen and then weighed were placed in series in the combustion tube, which was kept at 700 deg. C. during the run.

Before the experiment was begun the analysing train was thoroughly washed out with pure nitrogen in order to remove all air. The nitrogen used for this purpose was purified by passing it over hot copper, and through wash bottles containing barium hydroxide solution. The nitrogen thus purified gave zero test for both carbon dioxide and oxygen.

11. *Flushing the Furnace.*—In order to remove adsorbed gases from the glass pot and the insulating materials, the following procedure was adopted. The furnace was assembled as shown in Fig. 3 with the melting pot in place. The Gaede pump was started and the pressure in the furnace reduced to 0.02 mm. At the same time the current was started in the heating coil and the pot heated to a temperature several hundred degrees higher than that employed in the melting operation. The furnace was kept hot and the Gaede

pump in operation for several hours. Pure nitrogen was then admitted to the furnace chamber until atmospheric pressure was attained, after which the nitrogen was pumped out. This washing with nitrogen was repeated several times and the furnace finally allowed to cool while filled with nitrogen.

12. *Melting the Glass.*—When the nitrogen filled furnace was entirely cold, the water cooled dome was hoisted sufficiently to permit a weighed quantity (about 350 grams) of glass to be dropped into the melting pot, after which the dome was immediately lowered into place and the Gaede pump started. At the same time the resistor was heated to just below red heat, and after the pressure had fallen to 0.02 mm. the furnace was again flushed two times with pure nitrogen.

Finally, with a vacuum of 0.01 to 0.02 mm. in the furnace, it was sealed by closing a stop-cock, and the temperature of the pot was raised to about 1000 deg. C. and kept there for one hour. The pressure was then noted and the temperature of the pot allowed to drop to about 650 deg. C. after which pure nitrogen was admitted until atmospheric pressure had been reached.

With the resistor maintained at about 650 deg. C. the contents of the furnace were then pumped out through the analysing train and the furnace washed out with nitrogen, the washings being also pumped out through the analysing train. The furnace was finally allowed to stand full of pure nitrogen until time for the next experiment.

13. *The Results.*—The results obtained in four separate experiments, using pieces of the same block of glass, are shown in Table 1. It will be noticed that oxygen and carbon dioxide are present in solution in the glass to the extent of 0.1 per cent of its weight. Part, perhaps the greater part, of the carbon dioxide is present in the combined state as carbonate, and some of it would therefore be retained in the glass even under a vacuum of 0.02 mm.

TABLE 1

PER CENT BY WEIGHT OF OXYGEN AND OF CARBON DIOXIDE DISSOLVED IN A BARIUM-FLINT OPTICAL GLASS

GAS	WEIGHT PER CENT					MOLES PER LITER
	1	2	3	4	Mean	
O_2	0.078	0.092	0.074	0.086	0.08	0.07
CO_2	.017	.023	.031	....	.02	0.01

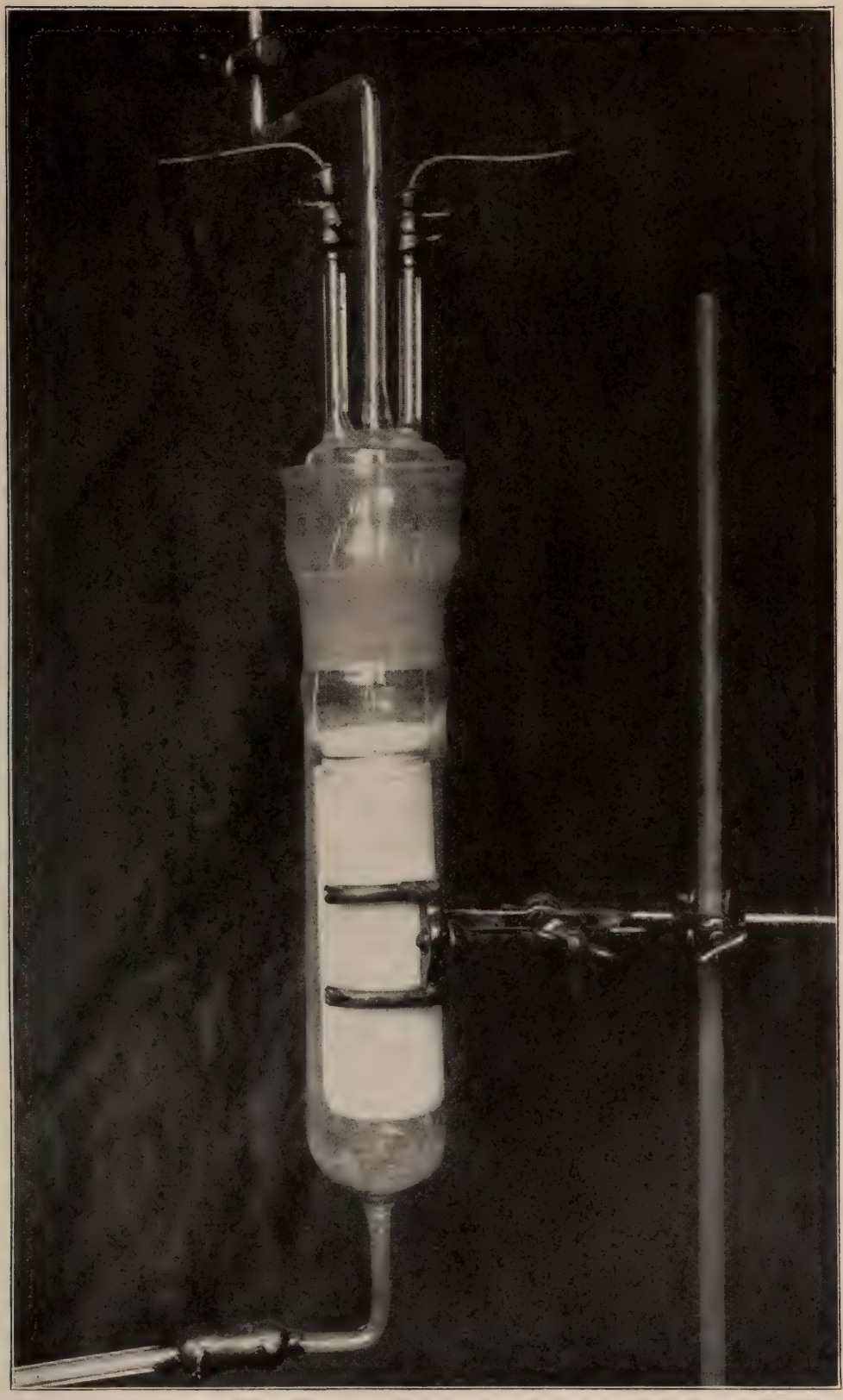

FIG. 6. APPARATUS FOR MEASURING AND ANALYSING THE DISSOLVED GASES IN GLASS

IV. A Special Apparatus for both Measuring and Analysing the Dissolved Gases in Glass

14. *Description of the Apparatus.*—The experiments described in the preceding section gave satisfactory evidence of the existence of dissolved oxygen and carbon dioxide in considerable amounts in finished glass. The apparatus and the method employed in these experiments were, however, rather cumbersome and complicated, and it was very difficult to make sure that no leakage of atmospheric gases into the evacuated furnace took place. The method, moreover, did not yield a measure of the total amount of the dissolved gases.

In order to eliminate these drawbacks a new vacuum furnace was designed, constructed entirely of glass and porcelain, which could readily be made perfectly gas tight, and which also permitted all of the gas evolved by the glass to be both measured and analysed. The final form of this apparatus is shown in Figs. 6 and 7.

The vacuum casing of the furnace consisted of a pyrex glass tube 5 cm. in diameter and 13 cm. high, provided with a ground glass stopper having a mercury seal at the joint. The melting pot was a cylindrical porcelain tube, 3 cm. in diameter and 13 cm. high. It was wound with platinum wire and slipped into a tightly fitting porcelain protecting tube. An outer more loosely fitting protecting tube completed this portion of the apparatus, which was suspended inside of the glass tube by means of two heavy copper leads which passed out through the capillary tubes, T_1 and T_2. The joint between these lead wires and the top of the capillary tubes was made tight by a rubber plug covered with a beeswax-rosin mixture.

15. *Determination of Free Volume of Furnace.*—For this purpose a calibrated 230 cu. cm. flask containing air at atmospheric pressure was attached at M and the stop-cock S_1 was closed. The furnace, containing the melting pot and its protecting tubes, was then evacuated to a pressure of 0.1 mm. of mercury and, after the connection to the pump had been closed, stop-cock S_1 was opened and the manometer reading again taken. The volume of the flask being known, and the change in pressure which occurred on connecting it to the evacuated apparatus, the free volume of the latter was calculated to be 475 cu. cm.

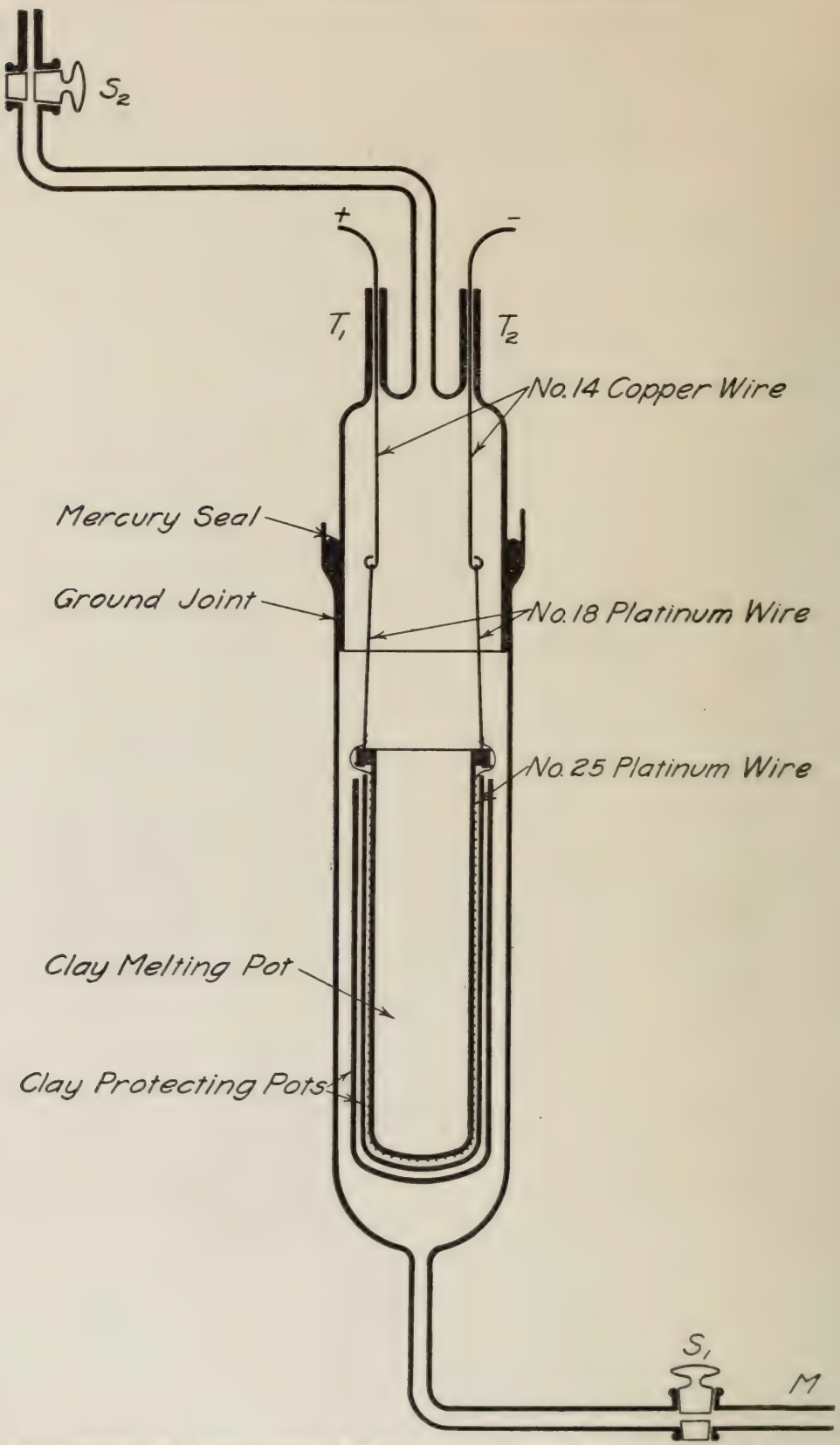

FIG. 7. DETAIL OF APPARATUS FOR MEASURING AND ANALYSING THE DISSOLVED GASES IN GLASS

16. *Experimental Procedure.*—The following procedure was employed in measuring and analysing the dissolved gases in glass. A weighed sample of glass—in some cases, 25 grams, in others, 50 grams,—was placed in the melting pot and the whole apparatus assembled as shown in the figure. With stop-cock S_1 closed, the apparatus was evacuated to a pressure of 0.1 mm. of mercury, and at the same time a sufficient current was passed through the heating wire to heat the pot and protecting tubes to about 400 deg. C., at which temperature no dissolved gas is given up by the glass. This preliminary heating and evacuating was necessary in order to remove adsorbed moisture from the porcelain. The connection to the pump was then closed and the whole apparatus allowed to stand for several hours in order to make sure that it was perfectly tight, this fact, of course, being indicated by an absolutely constant manometer reading.

Sufficient current was then passed through the heating coil to raise the temperature of the glass to 1400 deg. C. and the heating was continued till no more gas was evolved from the molten glass, as shown by a steady manometer reading. During this heating a blast of air was directed on the ground glass joint. The apparatus was then cooled to room temperature and the manometer reading was recorded. The free volume of the furnace being known, the total amount of gas evolved by the glass could be calculated. The total time required for a run was from two to three hours.

After the final manometer reading had been taken, the stop-cock leading to the manometer was closed, the manometer disconnected, and a small Orsat apparatus connected in its place. The tube M was then connected to an adjustable mercury reservoir, the mercury filling the tube completely up to the stop-cock. This stop-cock was opened and the furnace completely filled with mercury, all of the gas being driven out ahead of the mercury into the Orsat apparatus, where it was analysed for carbon dioxide and oxygen, any residual gas being considered nitrogen. The accuracy of the chemical analysis was about one per cent.

V. The Gas Content of Three Types of Commercial Glass

17. *A Barium Flint Optical Glass.*—This was the same type of glass as that used in the experiments described in Chapter III, but was obtained from the Pittsburgh Plate Glass Company, and may have differed somewhat in composition. Its index of refraction was given by Dr. Hostetter as "about 1.605, and its ν value, about 43.6."

Two experiments on 50 gram portions, and one on a 25 gram portion, of one block of glass gave total volumes of dissolved gases (measured under standard conditions) of 15.6, 14.3 and 8.04 cu. cm. respectively. The average is 15.3 cu. cm. for 50 grams of glass, amounting to 1.1 times the volume of the glass itself. Two samples of another block of the glass gave a volume of gas (under standard conditions) equal to half the volume of the glass. The history of the two blocks used in these experiments is not known. They were taken from a 25 lb. lot of cullet, and may have come from two entirely different melts. It is, of course, to be expected that the gas content of finished glass will depend very materially upon the melting and fining procedure which has been followed.

The gas from the second block of glass was analysed, and was found to consist of 25 per cent carbon dioxide and 75 per cent oxygen. If any nitrogen was present it was less than one per cent.

18. *A Light Flint Bulb Glass.*—The sample of glass used had the following composition according to the manufacturer's analysis:

Oxide . . .	SiO_2	PbO	Al_2O_3	CaO	Na_2O
Mole (per cent)	75.0	7.15	0.45	0.60	16.76

Two melts of 50 grams each were made and they gave respectively 3.2 and 3.5 cu. cm. of gas under standard conditions. The dissolved gas thus amounted to 0.2 times the volume of the glass itself. Analysis of the gas gave 58 per cent carbon dioxide, 24 per cent oxygen, and 18 per cent nitrogen. The density of the glass was 2.89.

19. *A Borosilicate Laboratory Glass.*—The glass investigated had approximately the following composition:

Oxide . . .	SiO_2	B_2O_3	As_2O_5	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O
Mole (per cent)	83.0	10.5	0.2	1.2	0.1	0.3	0.1	4.4	0.1

Twenty-five grams of glass gave, on melting, a volume of gas (under standard conditions) equal to 0.2 times the volume of the glass. On analysis the gas was found to consist of 26 per cent carbon dioxide, 37 per cent oxygen, and 37 per cent nitrogen.

TABLE 2

SUMMARY OF RESULTS ON THE AMOUNTS OF DISSOLVED GASES IN FINISHED GLASS

	VOLUME PER CENT S.T.P.				WEIGHT PER CENT				CONCENTRATION MOLES PER LITER		
GLASS	O_2	CO_2	N_2	Total	O_2	CO_2	N_2	Total	O_2	CO_2	N_2
Barium flint .1.....	83	27	<1	110	0.035	0.011	....	0.046	0.033	0.011	....
Barium flint .2.....	36	12	<1	48	.015	.0045	....	.020	.016	.005	....
Light flint.....	4.5	10	3	18	.0045	.014	0.0025	.021	.004	.010	0.003
Borosilicate.....	6	5	6	17	.0036	.0035	.0031	.010	.0028	.002	.0028
Water at 0 deg. C.									.0023	.080	.0010

20. *Discussion of the Results.*—The results obtained with the above three varieties of glass are summarized in Table 2. Owing to lack of data concerning the melting schedule and finishing operation used in the melts from which the samples studied originated, it is impossible to correlate the results obtained with the manufacturing procedure.

The quantity and nature of the gases present in the finished glass must obviously depend upon the batch composition and the melting and finishing procedures. The influence of the latter factor is probably responsible for the different results obtained with the different samples of the barium flint optical.

It is not probable that any appreciable quantities of gas are absorbed by the glass from the atmosphere of the furnace, except possibly in the case of glasses which are mechanically stirred for a long period. This conclusion seems to be borne out by the absence of nitrogen from the barium flint glass. The dissolved gas must therefore originate from the gases given off by the batch itself during the melting and fining processes.

At the end of the melting period, just before the fining operation begins, the glass usually contains numbers of small bubbles in which, owing to the high surface tension of molten glass, the gas is under a pressure greater than atmospheric. At the end of the fining opera-

tion the glass is therefore probably still somewhat supersaturated with gas, since, owing to its high viscosity, it cannot very rapidly give up this extra dissolved gas. The higher the finishing temperature, and the longer the glass is held at high temperatures, the smaller should be the amount of dissolved gas remaining in the finished glass. This conclusion seems to be borne out by the results obtained with the borosilicate glass, which is a glass requiring very high finishing and working temperatures. The great preponderance of acidic constituents in this glass may, however, be partially responsible for the small quantity of carbon dioxide found, since as shown by Niggli,* a good part of the dissolved carbon dioxide in glass is probably combined with the basic constituents.

* Niggli, Paul, "The Phenomena of Equilibria between Silica and the Alkali Carbonates," Jour. Amer. Chem. Soc., Vol. 35, 1706 (1913).

VI. The Significance of Dissolved Gases in Glass

21. *The Relation between Adsorbed and Dissolved Gas.*—It has long been recognized that glass in common with many other substances displays a strong tendency to adsorb, that is, to condense upon its surface, gases with which it is in contact.*

Adsorption may indeed be regarded as a type of solution in which the dissolved molecules do not penetrate below the surface layer of the adsorbent. Such a "surface solution" will therefore ordinarily be saturated when the surface of the adsorbent is covered with a layer of the adsorbed material one molecule deep and with its molecules close-packed laterally.†

Adsorption may sometimes be accompanied by a gradual penetration of the adsorbed material beneath the surface layer of the adsorbent, that is, it may be accompanied by ordinary or "volume" solution; but in the case of glass at low temperatures, such solution will probably be confined to the superficial layers. Adsorbed or superficially dissolved gases are thus to be distinguished from the dissolved gases studied in the present investigation, which are more or less uniformly disseminated throughout the whole mass of the glass. Langmuir‡ has found that water vapor is adsorbed and then slowly dissolved by glass. He also found that lamp bulbs when heated in vacuo evolved adsorbed carbon dioxide and nitrogen in addition to water vapor.

Recently Sherwood§ has devised a dynamic method for studying the gases evolved by glass when heated in vacuo to temperatures below its softening point. He found that adsorbed gases could be removed completely by heating to 200 deg. C. in vacuo and that the amount of such gases corresponded to a layer about one molecule

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* Cf. Guichard, M., "Sur les gaz dégagé des parois des tubes de verre." Bull. Soc. Chim. 100, 440 (1911).

† For a more detailed discussion of the relation between adsorption and solution see Washburn, E. W., "Introduction to the Principles of Physical Chemistry," Ed. 2, Chap. XXV, The McGraw-Hill Book Company, New York, 1921.

‡ Langmuir, I., "The Adsorption of Gases on Plane Surfaces of Glass, Mica and Platinum," Jour. Amer. Chem. Soc., Vol. 38, 2283-4 (1916); Ibid., Vol. 40, 1387 (1918).

§ Sherwood, R. G., "Gases and Vapors from Glass," Phys. Rev., Vol. 12, 448 (1918).

deep over the surface of the glass. On subsequent heating to 500 deg. C. a further evolution of gas occurred, which he attributed to "chemical reactions" occurring within the glass.

The well known jump in pressure within an exhausted glass vessel which occurs when it is "sealed off" and the subsequent deterioration of the vacuum with time has been studied by Shrader.* He concludes that: "The vacuum in sealed vessels deteriorates with time, rapidly at first, and then more slowly, and subsequent heating, even at temperatures lower than the heat-treating temperature, results in increase of pressure due to further liberation of the gases and vapors from the glass. No connection between different samples of the same glass or different glasses can be established. It is quite probable that there are variations in the properties of different samples of the same glass quite as great as the variations between different glasses of about the same grade."

There is every reason to believe that the dissolved gases in glass play an important role in the behavior described by Shrader. Certainly the jump in pressure which occurs during the sealing-off process can be ascribed to this source, and it seems entirely probable that gas-free glass would be superior in many respects to ordinary glass for the manufacture of high vacuum apparatus.

The adsorption of various gases by glass and their subsequent evolution with vacuum-heat treatment have been studied, particularly with respect to the production and maintenance of high vacua, by Ulrey† in a recent investigation which at the time of writing is available only in abstract. His conclusions in the main substantiate those already referred to, but the following may be mentioned:

(1) "Glass from which practically all absorbed gases have been removed by melting in vacuo subsequently reabsorbed gases from the atmosphere at room temperature."

(2) "At temperatures up to the softening point, diffusion of gases of the atmosphere through glass does not take place."

With reference to his first conclusion, a distinction should be drawn between absorbed or dissolved gases and adsorbed gases. The removal of the former by melting in vacuo does not, of course, affect

* Shrader, J. E., "Residual Gases and Vapors in Glass Bulbs," Phys. Rev., Vol. 13, 437 (1919).

† Ulrey, D., "Evolution and Absorption of Gases by Glass," Abstract in Phys. Rev., Vol. 14, 160 (1919).

the ability of the glass to adsorb gases from the atmosphere, the latter being an entirely independent process.

The evidence for his second conclusion not being available, its exact significance is not entirely clear. The fact that the atmospheric gases seem able to diffuse through quartz glass at comparatively low temperatures renders it not improbable* that a similar behavior might be exhibited by some of the ordinary commercial glasses under some circumstances, although doubtless to a considerably less extent.

22. *The Influence of Dissolved Gases upon the Properties and Behavior of Glass.*—Evolution of dissolved gas in the form of bubbles tends to occur whenever the pressure on glass, while in a fluid condition, is decreased. Such a decrease in pressure will occur during the manufacturing operation whenever there is a marked fall in the barometer, and it would be interesting to know whether there is any record of troubles with "seedy" glass accompanying periods of barometric depression.

A condition of reduced pressure with a consequent evolution of bubbles of gas also results from the strains set up by the contraction of the glass itself. If the outside of a mass of glass be allowed to solidify while the interior is still in a fluid condition, it is evident that the gradual solidification of the remaining glass must bring about a tension upon the still fluid portions; and this decrease in pressure will cause them to evolve their dissolved gases, with the consequent formation of a mass of bubbles in those portions of the glass which remain longest in the fluid condition. Some interesting examples of the formation of seed from this cause have been described by Williams.†

Still another instance of the occurrence of reduced pressure during manufacturing operations is met with in cases where the glass is "gathered" by suction, as in the case of the Owens machine. If the glass when it reaches the gathering machine is supersaturated with dissolved gases, the operation of gathering will evidently result in the formation of seed, some of which will not disappear again when the suction is released. If the glass at the moment of gathering is

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* Cf. Le Chatelier, "La Silice et les Silicates," p. 94, Hermann et Fils, Paris 1914; Mayer, E. C., "Leakage of Gases through Quartz Tubes," Phys. Rev., Vol. 4, 283 (1915).

† Williams, A. E., "Observations on the Formation of Seed in Optical Glass Melts," Jour. Amer. Ceramic Soc., Vol. I, 134 (1918).

undersaturated with the dissolved gases, as will be the case if it has been kept long enough at a sufficiently high temperature before reaching the gathering machine, the suction may still result in the momentary appearance of seeds, but these will be largely of the vacuum type and not permanent; if permanent, they will probably be exceedingly small, after the glass is finished.

Since both the appearance and disappearance of the seeds under these conditions is a process requiring a certain amount of time for the attainment of equilibrium, the viscosity of the glass, the time during which it is under the reduced pressure, and the subsequent cooling and annealing operations will evidently all have an influence on the final state of the glass as regards freedom from seeds. Seeds formed from glass which is supersaturated or practically completely saturated with dissolved gases cannot be removed by annealing, but seeds resulting from reduced pressure upon glass which is undersaturated with dissolved gases will disappear or be greatly reduced in size by proper annealing.

The complete story of the effect of dissolved gases upon the properties and behavior of glass must await the results of further investigation. It seems entirely probable that the presence of dissolved gases, and especially of microscopic seeds, may materially increase the tendency of the glass to devitrify, and the entire removal of this constituent might make it possible to obtain results which are not possible under ordinary conditions owing to the rapidity of devitrification. Certainly the known facts concerning the behavior of other supercooled solutions point in this direction.*

23. *The Use of Vacuum Furnaces in the Manufacture of Glass.* —The results described in the preceding pages are to be regarded as preliminary only. It is intended to continue the investigation not only for the purpose of determining the influence of dissolved gases upon the properties and behavior of glass, but also for the purpose of determining the practicability and value of a commercial process for the production of gas free glass.

The investigation of this subject was started early in 1918, and the results thus far attained indicate that a vacuum furnace process for the manufacture of certain types of glass is entirely feasible on

* In this connection see Germann, A. F. O., "The Devitrification of Glass, a Surface Phenomenon. The Repair of Crystallized Glass Apparatus." Jour. Amer. Chem. Soc., Vol. 43, 11 (1921).

an industrial scale, and that it offers a number of pronounced advantages over current methods. It eliminates entirely the fining operation as ordinarily understood, materially reduces the high finishing temperatures required with some glasses, produces in all cases a product absolutely free from even the smallest seeds, and these results suggest the possibility of considerably increasing the yield of perfect glass. Its main field of usefulness will probably be in the manufacture of certain types of optical glasses and of glass for high vacuum apparatus.

VII. Summary

24. *Summary of Results.*—The results of the investigation to date may be summarized as follows:

(1) All varieties of glass in the finished state contain dissolved gases.

(2) The amount of this dissolved gas is sufficient to cause the glass to effervesce violently if the pressure upon it be suddenly reduced while it is in a fluid condition.

(3) The amount and composition of the dissolved gas varies greatly with the type of glass and the detail of the melting and fining procedures.

(4) In the three types of industrial glass examined, the volume of the dissolved gases (measured under standard conditions) varied from 0.2 to 2 times the volume of the glass itself.

(5) Carbon dioxide, oxygen, and nitrogen were found in varying amounts in the gas.

In addition, as a result of this experimental work, a convenient apparatus for measuring and analysing the dissolved gases was developed, and an improved type of vacuum furnace for the manufacture of gas-free glass was constructed.

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